



October 21, 2016

Tom Moe USS Corporation P.O. Box 417 8771 Park Ridge Dr Mountain Iron, MN 55768

RE: Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1276472

#### Dear Tom Moe:

Enclosed are the analytical results for sample(s) received by the laboratory on October 06, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current, applicable TNI/NELAC standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Melisa M Woods

Massia Wirds

melisa.woods@pacelabs.com

**Project Manager** 

**Enclosures** 

cc: Cory Hertling Terri Sabetti, NTS







#### **CERTIFICATIONS**

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1276472

Virginia Minnesota Certification ID's

315 Chestnut Street, Virginia, MN 55792

Alaska Certification UST-107 Alaska Certification UST-107

Alaska Certification #MN01084

Arizona Department of Health Certification #AZ0785

Minnesota Dept of Health Certification #: 027-137-445

North Dakota Certification: # R-203

Wisconsin DNR Certification #: 998027470 WA Department of Ecology Lab ID# C1007

Nevada DNR #MN010842015-1

Oklahoma Department of Environmental Quality

**Duluth Minnesota Cerification ID's** 

4730 Oneota St., Duluth, MN 55807

4730 Offeold St., Dulutti, WiN 55607

Minnesota Dept of Health Certification #: 027-137-152

Wisconsin DNR Certification #: 999446800

North Dakota Certification #: R-105

#### **REPORT OF LABORATORY ANALYSIS**



#### **SAMPLE SUMMARY**

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1276472

| Lab ID     | Sample ID         | Matrix | Date Collected | Date Received  |
|------------|-------------------|--------|----------------|----------------|
| 1276472001 | SD 001 (Seep 020) | Water  | 10/06/16 11:30 | 10/06/16 13:05 |

#### **REPORT OF LABORATORY ANALYSIS**



#### **SAMPLE ANALYTE COUNT**

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1276472

| Lab ID     | Sample ID         | Method               | Analysts | Analytes<br>Reported | Laboratory |
|------------|-------------------|----------------------|----------|----------------------|------------|
| 1276472001 | SD 001 (Seep 020) | EPA 1664A TPH (1999) | BT1      | 1                    | PASI-DUL   |
|            |                   | USGS I-3765          | JJH      | 1                    | PASI-V     |
|            |                   | EPA 300.0            | DMB      | 1                    | PASI-V     |



#### **ANALYTICAL RESULTS**

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1276472

Date: 10/21/2016 07:55 AM

| Sample: SD 001 (Seep 020)          | Lab ID:     | Collected: 10/06/16 11:30 |                 |        | Received: 10/06/16 13:05 Matrix: Water |                |                |            |      |
|------------------------------------|-------------|---------------------------|-----------------|--------|--|----------------|----------------|------------|------|
| Parameters                         | Results     | Units                     | Report<br>Limit | MDL    | DF                                     | Prepared       | Analyzed       | CAS No.    | Qual |
| 1664 SGT-HEM, TPH                  | Analytical  | Method: EPA               | 1664A TPH       | (1999) |  |                | _              |            |      |
| Total Petroleum Hydrocarbons ND mg |             | mg/L                      | 3.0             | 1.0    | 1                                      | 10/19/16 11:34 |                |            |      |
| USGS I-3765 TSS                    | Method: USG | S I-3765                  |                 |        |  |                |                |            |      |
| Total Suspended Solids 1.2 mg/L    |             | mg/L                      | 1.0             | 1.0    | 1                                      |                | 10/13/16 11:29 |            |      |
| 300.0 IC Anions 28 Days            | Analytical  | Method: EPA               | 300.0           |        |  |                |                |            |      |
| Sulfate                            | 965         | mg/L                      | 20.0            | 10.0   | 10                                     |                | 10/19/16 15:51 | 14808-79-8 |      |



#### **QUALITY CONTROL DATA**

EPA 1664A TPH (1999)

1664 SGT-HEM, TPH

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1276472

QC Batch: 97671

QC Batch Method: EPA 1664A TPH (1999)

Associated Lab Samples: 1276472001

METHOD BLANK: 386894 Matrix: Water

Associated Lab Samples: 1276472001

Blank Reporting

Parameter Units Result Limit MDL Analyzed Qualifiers

Total Petroleum Hydrocarbons mg/L ND 3.0 1.0 10/19/16 11:34

Analysis Method:

Analysis Description:

LABORATORY CONTROL SAMPLE: 386895

Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers 76 Total Petroleum Hydrocarbons mg/L 20 15.3 64-132

MATRIX SPIKE SAMPLE: 386896

Date: 10/21/2016 07:55 AM

1276472001 MS MS Spike % Rec Parameter Units Result Conc. Result % Rec Limits Qualifiers ND 64-132 Total Petroleum Hydrocarbons 20 16.9 84 mg/L

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



#### **QUALITY CONTROL DATA**

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1276472

QC Batch: 97175 Analysis Method: USGS I-3765

QC Batch Method: USGS I-3765 Analysis Description: USGS I-3765 Total Suspended Solids

Associated Lab Samples: 1276472001

METHOD BLANK: 383958 Matrix: Water

Associated Lab Samples: 1276472001

Blank Reporting
Parameter Units Result Limit MDL Analyzed Qualifiers

Total Suspended Solids mg/L ND 1.0 1.0 10/13/16 11:28

LABORATORY CONTROL SAMPLE: 383959

Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers **Total Suspended Solids** mg/L 239 234 98 80-120

SAMPLE DUPLICATE: 383960

1276536001 Dup Max **RPD RPD** Parameter Units Result Result Qualifiers 270 280 4 10 Total Suspended Solids mg/L

SAMPLE DUPLICATE: 383961

Date: 10/21/2016 07:55 AM

ParameterUnits1276511001 ResultDup ResultRPDMax RPDQualifiersTotal Suspended Solidsmg/L306318410

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



#### **QUALITY CONTROL DATA**

EPA 300.0

300.0 IC Anions

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1276472

Date: 10/21/2016 07:55 AM

QC Batch: 97707 Analysis Method: QC Batch Method: EPA 300.0 Analysis Description:

Associated Lab Samples: 1276472001

METHOD BLANK: 387038 Matrix: Water

Associated Lab Samples: 1276472001

ParameterUnitsBlank Reporting ResultReporting LimitMDLAnalyzedQualifiersSulfatemg/LND2.01.010/19/16 14:23

LABORATORY CONTROL SAMPLE: 387039

Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers Sulfate mg/L 50 52.5 105 90-110

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 387040 387041 MS MSD

1276472001 Spike Spike MS MSD MS MSD % Rec Max Parameter Units Result Conc. Conc. Result Result % Rec % Rec Limits RPD RPD Qual Sulfate 500 90-110 0 20 mg/L 965 500 1460 1460 100 100

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 387042 387043

| Parameter | Units | 1277311010<br>Result | Spike<br>Conc. | Spike<br>Conc. | MS<br>Result | MSD<br>Result | MS<br>% Rec | MSD<br>% Rec | % Rec<br>Limits | Max<br>RPD RPD |   |
|-----------|-------|----------------------|----------------|----------------|--------------|---------------|-------------|--------------|-----------------|----------------|---|
| Sulfate   | mg/L  | 2.1                  | 50             | 50             | 53.3         | 53.3          | 102         | 102          | 90-110          | 0 2            | ) |

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Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

#### **REPORT OF LABORATORY ANALYSIS**



#### **QUALIFIERS**

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1276472

#### **DEFINITIONS**

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

**DUP - Sample Duplicate** 

**RPD - Relative Percent Difference** 

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

#### **LABORATORIES**

Date: 10/21/2016 07:55 AM

PASI-DUL Pace Analytical Services - Duluth
PASI-V Pace Analytical Services - Virginia



#### **QUALITY CONTROL DATA CROSS REFERENCE TABLE**

Project: USS MinnTac NPDES-TB Wk1

Pace Project No.: 1276472

Date: 10/21/2016 07:55 AM

| Lab ID     | Sample ID         | QC Batch Method      | QC Batch | Analytical Method | Analytical<br>Batch |
|------------|-------------------|----------------------|----------|-------------------|---------------------|
| 1276472001 | SD 001 (Seep 020) | EPA 1664A TPH (1999) | 97671    |                   |                     |
| 1276472001 | SD 001 (Seep 020) | USGS I-3765          | 97175    |                   |                     |
| 1276472001 | SD 001 (Seep 020) | EPA 300.0            | 97707    |                   |                     |

Required Client Information:
Company: USS Corporation
Address: P.O. Box 417 Mt. Iron, MN 55768 ITEM # equested Due Date: SD 001 (Seep 020) Sample Ids must be unique One Character per box. (A-Z, 0-9/, -) SAMPLE ID Fax MATRIX
Divinking Water
Water
Waste Water
Product
Soil/Soild
Oil
Wipe
Air
Chner
Tissue Required Project Information:
Report To: Tom Moe
Copy To: Purchase Order #:
Project Name: NPDES-TB Wk1 Project #: Section B TS P WW DW CODE MATRIX CODE (see valid codes to left) fund restate SAMPLE TYPE (G=GRAB C=COMP) 25-20/04; 11/32 9-10/ DATE START COLLECTED DATE CHAIN-OF-CUSTODY / Analytical Request
The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields 

NO 11 1 1276472 S 10-6-16 1340 TIME SAMPLE TEMP AT COLLECTION Section C Invoice Information: # OF CONTAINERS Pace Quote: Pace Project Manager: Attention: Pace Profile #: Company Name: Address: Unpreserved H2SO4 **EONH** Preservatives HCI NaOH Na2S2O3 heather.zika@pacelabs.com, Methanol Other And the street V, V TSS,SQ4 TRPH 1664 CLIENTO USS CORP ENE MIN 10-6-16 13.05 3.7 Residual Chlorine (Y/N) ate: 10/20/16 2

SIGNATURE of SAMPLER: PRINT Name of SAMPLER:

lan maxic

and water

DATE Signed:

107676

TEMP in C

Received on

ice (Y/N) Custody Sealed

Cooler (Y/N) Samples Intact (Y/N)

Sample Condition Upon Receipt Form Page 1 of 1

Document No.: Issuing Authority:
F-VM-C-001-Rev.09 Pace Virginia, Minnesota Quality Office

|   | <u> </u>    |                | _            | WO#:1276472  PM: MMW Due Date: 10/20/16                                    |
|---|-------------|----------------|--------------|--|
| Courier: Fed Ex UPS  Commercial Pace  | USPS        | _              | Client       | CLIENT: USS CORP   |
| Tracking Number:  | Othe        | r:             | ··           |  |
| Custody Seal on Cooler/Box Present? Yes   | <b>∑</b> No | Seals          | Intact?      | Yes No Optional: Proj. Due Date: Proj. Name:                               |
| Packing Material: Bubble Wrap Dubble  | Bags 🔲 r    | Vone           | Other:_      | Temp Blank? 📝 Ŷes 🗀 No   |
| Thermometer Used: 🔲 140792808   | Type of     | fice:          | Wet          | Talue None Ramoles on iso speliar  |
| Cooler Temp Read °C: 3.4 Cooler Temp  | Corrected   | ۰c.            | <u> </u>     | Somples office, cooling process has be                                     |
| emp should be above freezing to 6°C Correction Fa   | ctor:       | 6-3            | Date ar      | Biological Tissue Frozen? Yes No de Initials of Person Examining Contents: |
| Chain of Custody Present?   | € Yes       |                |              | Comments:  |
| Chain of Custody Filled Out?  | ✓ Yes       | □No            | □N/A         |  |
| Chain of Custody Relinquished?  | Z Yes       | N <sub>0</sub> |              |  |
| Sampler Name and Signature on COC?  | Z) Yes      | □No            |              | 4.   |
| Samples Arrived within Hold Time?   | ZYes        |                |              | 5.   |
| Short Hold Time Analysis (<72 hr)?  | Yes         | ZNO            |              | 6.   |
| Rush Turn Around Time Requested?  | Yes         | 7<br>          | □N/A         | 7.   |
| Sufficient Volume?  | Yes         | □No            | □N/A         |  |
| Correct Containers Used?  | Z Yes       | □No            | □N/A         | 9.   |
| -Pace Containers Used?  | Yes         | _<br>□No       | □N/A         |  |
| Containers Intact?  | . □⁄Yes     | □No            | □n/a         | 10.  |
| Fiftered Volume Received for Dissolved Tests?   | Yes         | □No            | Z N/A        | 11. Note if sediment is visible in the dissolved containers.               |
| Sample Labels Match COC?  | Yes         | □No            | □N/A         | 17.  |
| -Includes Date/Time/ID/Analysis Matrix:   | <u>٠٠</u> ٠ |                |              |  |
| All containers needing acid/base preservation will be checked and documented in the pH logbook. | Yes         | □No            | N/A          | See pH log for results and additional preservation documentation           |
| Head space in Methyl Mercury Container  | Yes         | □No            | ØN/A         | 13.  |
| Head space in VOA Vials ( >6mm)?  | □Yes        | □No            | ON/A         | 14,  |
| Trip Blank Present?   | Yes         | □No            | ØN/A         | 15.  |
| Frip Blank Custody Seals Present?   | □Yes        | □No            | <b>Ø</b> N/A |  |
| Pace Trip Blank Lot # (if purchased):   |             | <u> </u>       |              |  |
| IENT NOTIFICATION/RESOLUTION  |             |                |              | Field Data Required? Yes No  |
| Person Contacted:   |             |                | D            |  |
| Comments/Resolution:  |             |                | <del></del>  | ite/Time:  |
| 9   |             |                |              |  |
|   |             |                |              |  |
|   |             |                |              |  |
|   |             |                |              |  |
| CAL WAIVER ON FILE Y N  | <del></del> | Teres          |              |  |
| nn()  | , ,         | IEMP!          | KATURI       | WAIVER ON FILE Y N   |
| ject Manager Review   | 1/1         | _ /            | >            | 10/10/10   |

Page 12 of 14

# Item Virginia, MN 55792 Phone (218) 742-1042 Report To: Melisa M Woods 315 Chestnut Street Workorder: 1276472 Cooler Temperature on Receipt Pace Analytical Virginia Received at: Intra-Regional Chain of Custody Transfers Sample ID SD 001 (Seep 020) Released By Workorder Name: NPDS-TB Wk1 Type Sample PS ကိ Collect Date/Time 10/6/2016 11:30 Date/Time Send To Lab: 10/6/10 17/5 Duluth, MN 55807 Phone (218) 727-6380 Pace Analytical Duluth 4730 Oneota Street e16/16/16/cr **Custody Sea** Lab ID 1276472001 Received By Water Matrix HCL Preserved Containers Received on loe Owner Received Date: 10/6/2016 ð Date/Time 6 6 EPA 1664A TPH (1999) × Z Requested Analysis Due Date: 10/20/2016 Samples Intact Comments

LAB USE ONLY

Q

ç

Z

<sup>\*\*\*</sup>In order to maintain client confidentiality, location/name of the sampling site, sampler's name and signature may not be provided on this COC document. This chain of custody is considered complete as is since this information is available in the owner laboratory.

# ace Analytical <sup>®</sup>

## Document Name:

### Sample Condition Upon Receipt Form

Document No.:

F-DUL-C-001-Rev.01

Document Revised: 22Jan2016 Page 1 of 1

Issuing Authority:

Pace Virginia, Minnesota Quality Office

| Sample Condition Upon Receipt Client Name:   |                           |               | Project #          | :   |
|--|---------------------------|---------------|--------------------|---|
| Courier: Fed Ex UPS  | _                         |               | Client             |   |
| Tracking Number:   |                           |               |                    |   |
| Custody Seal on Cooler/Box Present? XYes   | □No                       | Seals I       | _ ′                | Yes No Optional: Proj. Due Date: Proj. Name:  |
| Packing Material: Subble Wrap  | oble Bags No              | one L         | Other:             | Temp Blank? Yes No  |
| Thermometer Used: 🔀 B00051   | Type of I                 | ce: 🔀         | Øvet [             | Blue None samples on ice, cooling process has begun                                     |
| Cooler Temp Read °C: 1.5 Cooler Temp should be above freezing to 6°C Correction            | emp Corrected °C          | : <u>0.</u> ° | Date and           | Biological Tissue Frozen?  Yes No NA  Initials of Person Examining Contents:  Comments: |
| Chain of Custody Present?  | <b>⊠</b> Yes              | □No           | □N/A               | 1.  |
| Chain of Custody Filled Out?   | ∑NYes                     | □No           | □N/A               | 2.  |
| Chain of Custody Relinquished?   | <b>Z</b> ¶⁄es             | □No           | □n/A               | 3.  |
| Sampler Name and Signature on COC?   | □Yes                      | □No           | N/A                | 4.  |
| Samples Arrived within Hold Time?  | Yes                       | □No           | □N/A               | 5.  |
| Short Hold Time Analysis (<72 hr)?   | <b>∕</b><br><b>∑</b> oves | □No           | □N/A               | 6.  |
| Rush Turn Around Time Requested?   | Yes                       | ₩No           | N/A                | 7.  |
| Sufficient Volume?   | <b>∕</b> es               | □No           | <br>□n/a           | 8.  |
| Correct Containers Used?   | Yes                       | □No           | N/A                | 9.  |
| -Pace Containers Used?   | es                        | —<br>□No      | □n/A               |   |
| Containers Intact?   | Yes                       | □No           | □N/A               | 10.   |
| Filtered Volume Received for Dissolved Tests?  | □Yes                      | □No           | <br><b>[20</b> ]/A | 11. Note if sediment is visible in the dissolved containers.                            |
|  | W 7. Dres                 | □No           | □N/A               | 12.   |
| -Includes Date/Time/ID/Analysis Matrix:  | <u> </u>                  |               |                    |   |
| All containers needing acid/base preservation wi checked and documented in the pH logbook. | ∥ be ∐Yes                 | □No           |                    | See pH log for results and additional preservation documentation                        |
| Headspace in Methyl Mercury Container  | ∐Yes                      | □No           | □n/A               | 13.   |
| Headspace in VOA Vials ( >6mm)?  | □Yes                      | □No           | □n/a               | 14.   |
| Trip Blank Present?  | ∐Yes                      | □No           | <b>□</b> N/A       | 15.   |
| Trip Blank Custody Seals Present?  | ∐Yes                      | □No           | <b>□</b> N/A       |   |
| Pace Trip Blank Lot # (if purchased):  |                           |               |                    |   |
| CLIENT NOTIFICATION/RESOLUTION   |                           |               |                    | Field Data Required? Yes No   |
| Person Contacted:  |                           |               |                    | Date/Time:  |
| Comments/Resolution:   |                           |               |                    |   |
|  |                           |               |                    |   |
| FECAL WAIVER ON FILE Y N   | $\widehat{}$              | TEM           | 1PERATU            | RE WAIVER ON FILE Y N   |
| Project Manager Review: W  | ME                        | -             |                    | Date: 10-7-16   |

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers)